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Application No. 09/926,513

Please insert the following paragraph for the paragraph appearing at page 1, lines 12-24, with a marked-up copy of the amended paragraph appearing in an Appendix attached to this reply:

B3 Ceramic sintered products based on aluminum oxide (Al_2O_3), particularly corundum ($\alpha\text{-Al}_2\text{O}_3$), are widely used due to the advantageous chemical and oxidative resistance of these products, in particular in the latter modification. This applies both to dense sintered products (for example, as tool material or wear-resistant machine parts), and to porous components (for example, as catalyst carriers or as filtering material). While dense structures having crystal sizes $> 2\mu\text{m}$ have been known for a long time, it has been possible to produce submicron structures only since the mid-80s by new sol/gel processes and since the beginning of the 90s as a result of the availability of finer crystalline corundum powders (grain size $\geq 150\text{ nm}$). Since then, the development of more and more finely structured sintered structures has been a priority goal for ceramic material development, both in the field of dense sintered products with the goal of greater hardness and wear resistance, and in the field of porous materials, e.g., for ultrafiltration membranes. Future advances are determined decisively by further development of more and more fine-grained raw materials.

Page 9, line 1, change "Description of the Invention" to --SUMMARY OF THE INVENTION--.

B4 Please insert the following paragraph for the paragraph appearing at page 10, lines 3-6, with a marked-up copy of the amended paragraph appearing in an Appendix attached to this reply:

B5 The ~~joint task of the~~ present inventions relates to a process for the production of chlorine-free nanocorundum of various porosities as an end or intermediate product, whereby the intermediate

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product can be processed further according to the invention to other products according to the invention.

Page 11, line 9 insert the following paragraphs:

The present invention is directed to a process for producing redispersible nanocorundum with an average particle size $D_{50} < 100$ nm with addition of nuclei that promote transformation to corundum in subsequent annealing, comprising:

(a) dissolving in a liquid medium or processing in a liquid medium to a sol, as starting materials, chlorine-free inorganic precursors;

(b) hydrolyzing the solution or the sol of (a) through the addition of a base in a mole ratio of base:precursor of 1 to 3;

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(c) aging the hydrolyzed solution or sol of (b) at temperatures between 60 and 98°C for 1 to 72 hours;

(d) subsequently drying the aged solution or sol of (c) followed by calcination at temperatures between 350 and 650°C for converting hydrolyzed precursor into a semiamorphous intermediate phase and ultimately into transitional aluminum oxides; and

(e) performing further annealing by increasing temperature to $\leq 950^\circ\text{C}$ for converting product of (d) into corundum phase.

The present invention is also directed to a process for producing redispersible nanocorundum with an average particle size $D_{50} < 100$ nm with addition of nuclei that promote transformation to corundum in subsequent annealing, comprising:

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(a) dissolving in a liquid medium or processing in a liquid medium to a sol, as starting materials, organic precursors;

(b) hydrolyzing either (i) with excess water through addition of the precursor solution or the precursor sol of (a) to water at a mole ratio of water:precursor > 3 , and with addition of an acid that leads to $\text{pH} = 3-5$, or (ii) through addition of an amount of water restricted to a mole ratio of water:precursor ≤ 3 to the precursor solution or precursor sol of (a) that are to be mixed with complex-forming ligands;

(c) aging the hydrolyzed solution or sol of (b) at temperatures of $\leq 50^\circ\text{C}$ within 5 hours, and subsequently aging at temperatures of 80 to 98°C within 1 to 24 hours;

(d) subsequently drying the aged solution or sol of (c) followed by calcination at temperatures between 350 and 650°C for converting the hydrolyzed precursor into a semiamorphous intermediate phase and then to transitional aluminum oxides; and

(e) performing further annealing by increasing temperature to $\leq 950^\circ\text{C}$ for converting product of (d) into corundum phase.

The calcination can be carried out at temperatures of 400 to 600°C for 0.5 to 2 hours, and the further annealing for formation of corundum can be carried out by a temperature increase to $650 - 900^\circ\text{C}$ for 0.5 to 1 hours.

The at least one of the transitional aluminum oxides and corundum can be ground.

The grinding of the at least one of the transitional aluminum oxides and corundum can be carried out in an organic liquid.

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After the aging of the hydrolyzed solution or sol, a gel formation or a liquid shaping can be carried out, subsequently the drying, calcination and annealing can take place and after the annealing a sintering can be carried out at temperatures above the corundum formation temperature.

The present invention is also directed to nanocorundum powders comprising a close particle size distribution in low nanometer range, comprising a narrow width of size distribution of isometrically formed particles $D_{84} < 150$ nm, less than 0.05% by weight chlorine, at least 60% α -aluminum oxide, and the powders are redispersible.

The present invention is also directed to a process for the production of sintered corundum products in a form of dense or porous compact bodies, layers or granulates, comprising sintering nanocorundum powders according to the present invention at temperatures $\leq 1450^\circ\text{C}$ to form granulate or sintered corundum bodies having an average grain size of $\leq 0.6 \mu\text{m}$.

The present invention is also directed to a process for coating a porous or dense metallic substrate wherein particles of the hydrolyzed sol or particles of a suspension of nanocorundum produced according to the present invention are electrophoretically deposited on the metallic substrate, and subsequently subjected to annealing.

The present invention is also directed to a process for the production of sintered porous or dense corundum layers wherein the aged the hydrolyzed solution or sol of (c) according to present invention is applied to a substrate and afterwards the drying, calcination and annealing are carried out.

After the aging of the solution or the sol, the material can be deposited on a substrate with gel formation.

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After the annealing for converting into the corundum phase, a sintering can be carried out at temperatures above the corundum formation temperature.

After annealing, at least one further coating and at least one further annealing can be carried out.

The present invention is also directed to Al_2O_3 sintered products, produced according to the present invention, wherein through annealing at 650 to 1250°C, there is a phase composition of more than 80% corundum and an average pore size of 10 - 100 nm with a porosity of $\geq 30\%$ by volume.

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com The present invention is also directed to dense sinter corundum layers, produced according to the present invention, on a materially different type of substrate, in which through sintering at a temperature of $\leq 1250^\circ\text{C}$ there is an average grain size of $\leq 0.5 \mu\text{m}$.

The present invention is also directed to a process for producing nanoporous Al_2O_3 sintered products comprising:

(a) dissolving in a liquid medium or processing in a liquid medium to a sol, as starting materials, chlorine-free inorganic precursors;

(b) hydrolyzing the solution or the sol of (a) through the addition of a base in a mole ratio of base:precursor of 1 to 3;

(c) aging the hydrolyzed solution or sol of (b) at temperatures between 60 and 98° C for 1 to 72 hours; and

(d) subsequently drying followed by calcination at temperatures between 350 and 750°C for converting hydrolyzed precursors into aluminum oxide.

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The present invention is also directed to a process for producing nanoporous Al_2O_3 sintered products, comprising:

(a) dissolving in a liquid medium or processing in a liquid medium to a sol, as starting materials, organic precursors;

(b) hydrolyzing either (i) with excess water through addition of the precursor solution or the precursor sol of (a) to water at a mole ratio of water:precursor > 3 , and with addition of an acid that leads to $\text{pH} = 3-5$, or (ii) through addition of an amount of water restricted to a mole ratio of water:precursor ≤ 3 to the precursor solution or precursor sol of (a) that are to be mixed with complex-forming ligands;

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(c) aging the hydrolyzed solution or sol of (b) at temperatures of $\leq 50^\circ\text{C}$ within 5 hours, and subsequently aging at temperatures of 80 to 98°C within 1 to 24 hours; and

(d) subsequently drying the aged solution or sol of (c) followed by calcination at temperatures between 350 and 750°C for converting hydrolyzed precursors into aluminum oxide.

The aged solution or the sol can be applied to a substrate and afterwards the drying and calcination can be carried out.

The gel formation can occur upon application to a substrate.

Nuclei of a transitional aluminum oxide can be added to the solution or to the sol.

The present invention is also directed to a process for coating a porous or dense metallic substrate, wherein particles of hydrolyzed sol or particles of a suspension of nano porous aluminum oxide according to the present invention are electrophoretically deposited on the metallic substrate.

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After the electrophoretic deposit of the particles, a heat treatment can be carried out at temperatures of 350 - 750°C.

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The present invention is also directed to nanoporous Al_2O_3 sintered products, produced according to the present invention in which there is an average pore diameter in the range between 0.5 and 2.5 nm at a porosity of $\geq 30\%$ by volume.

Page 11, delete the paragraph appearing at lines 10 and 11.

Page 11, line 12, insert: ~~1~~ DETAILED DESCRIPTION OF THE INVENTION---

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Please insert the following paragraph for the paragraph appearing at page 24, lines 1-6, with a marked-up copy of the amended paragraph appearing in an Appendix attached to this reply:

A technologically relevant criterium for the actual redispersibility of a nano corundum powder produced by annealing is its usability in a shaping process low in defects and with a low sintering temperature giving dense compacts from the shaped bodies. In an example for the production of dense sintered corundum products of nano corundum powder, particular importance was, moreover, focused on a high purity of the processes and products, any doping to promote the dense sintering or to limit the grain growth was abandoned.